1116

DETERMINATION OF THE SOLIDS IN MILK BY A LACTOMETRIC METHOD AT 102° F.[1]

PAUL D. WATSON

Eastern Utilization Research and Development Division, Agricultural Research Service, USDA, Washington, D. C.

Formulas for calculating the percentage of total solids and solids-not-fat in milk have been studied in the United States and abroad for many years. More than 60 different equations for computing the relation between the fat content and specific gravity and the solids of milk have been submitted by investigators. These equations appear to give reliable estimates of milk solids only when applied to data from which they were derived. Many researchers have substituted constants and corrections in the basic equations, in order to make the computed values agree with the experimental ones. The different constants, corrections, and divergencies in the calculated values have caused confusion and doubt concerning the fundamental reliability of the method. Comprehensive reviews and studies of the various formulas and techniques are available *(7, 8, 9, 10, 20)*.

The lag in the development of a more satisfactory method may be due to the emphasis that has been placed for many years on the fat content of milk as the basis for paying producers. The growing importance of the nonfat milk solids in the economy of the dairy industry has made it urgent to have a quick and economical method for determining milk solids, which would be comparable in speed and accuracy to the Babcock test for fat. Hence, an intensive study was made to develop a practical method for determining milk solids, which would avoid the difficulties previously encountered.

This method has been reported *(18)*. In the present paper, the sources of error, experimental results, and lactometer design are considered in more detail.

FORMULA FOR CALCULATION OF MILK SOLIDS

An algebraic equation was derived from the theoretical equation *(19)* for the calculation of the solids from the lactometer and the Babcock fat determinations. The constant (0.31) of the original formula was increased to 0.4 to compensate

[1] This work was done with funds delegated by the Agricultural Marketing Service.

for a change in the calibration of the lactometers, which was made later by the National Bureau of Standards:

$$\% \text{ T.S.} = 1.33 \text{ F} + \frac{273\text{L}}{\text{L} + 1000} - 0.40$$

F = Babcock fat % L = Lactometer reading in degrees

The nonfat solids may be found by using 0.33 F instead of 1.33 F, or by subtraction of the fat percentage from the total solids percentage. The total solids of skimmilk samples can be calculated by dropping the constant (0.40) from the formula.

SOURCES OF ERROR

A review of previous methods has indicated that unsatisfactory results are owing largely to two causes. One error is the indefinite physical state of milk fat at 60° F., the temperature in general use for the lactometer readings. The uncertain degree of solidification of fat at this temperature makes it unlikely that consistent values can be obtained. That the fat should be in a uniform physical state for the test has been known for many years, but solution of the problem has been slow. Various workers have preheated the milk samples to 104° F., or even as high as 113° F., and then cooled them to 60° or 86° F. before reading the lactometer *(2, 11, 17)*.

A second error is the general use of unsuitably designed and inaccurately calibrated lactometers. Many in use have not been tested for accuracy and have not been satisfactorily checked; few have been certified for accuracy by the National Bureau of Standards.

The literature indicates that it is common practice to check the calibration of lactometers in solutions of acid, salts, or sugars, which have a surface tension considerably higher than that of milk. A survey of commercial lactometers indicated none was entirely suitable for accurate milk-solids determinations. The practical types were so coarsely graduated that readings of the required precision could not be made, and the finely graduated instruments were too fragile for general use. Almost all types examined were found to have inaccurate scales, compared with lactometers which had been calibrated by the National Bureau of Standards. Invariably, no mention is made of any correction for differences of surface tension and of the meniscus, which can cause appreciable errors in evaluating the various formulas for calculating milk solids. These difficulties can be avoided by checking the calibration of a lactometer with milk, by means of specific gravity bottles. The use of normal milk is impractical owing to fat rising, but skimmilk and homogenized milk are suitable.

Since the percentage of fat has a marked effect on the specific gravity of milk, it is used with the lactometer reading in formulas for the calculation of total solids. It is known that Babcock tests for fat may differ by 0.1% or more in comparative tests by different technicians *(4, 12)*. An error of 0.1% results in a difference of 0.13% in the calculated total solids, and it has been held that an excessive degree of precision in the lactometer reading is not justified. The necessity for close temperature regulation has been ignored for the same reason.

Consequently, some researchers have questioned the need for the sensitive type of lactometer with a large bulb and a scale reading closer than 0.5 of a lactometer degree. Herrington *(8)* has pointed out that an error of 0.08° Quevenne is as important as an error of 0.1% of fat in the calculation of the nonfat solids of milk.

Roeder *(16)* made a comprehensive study of the sources of error in the total solids method in which the lactometer and Gerber fat values were used with the Fleischmann formula. The largest single source of error was attributed to variations in the specific gravity of the milk constituents. He estimated that a total possible theoretical error of $\pm$ 0.318% exists, but that the limits of error must be assumed to be between $\pm$ 0.15% total solids to allow for the possibility of a compensation of errors.

DEVELOPMENT OF METHOD

Consideration of the temperature problem has led to the conclusion that the lactometer method can be made more practical by eliminating the cooling step. which has been recommended by others. The new procedure consists of heating the milk samples to a temperature at which the milk fat is in a uniform liquid state and then reading the lactometer at this temperature. The temperature must be high enough to insure fat liquidity, but not so high that other physical changes might occur to affect the specific gravity of the milk. Experiments showed that consistently reproducible results can be obtained if the temperature is 102° F. (39° C.).

No provision was made for temperature variations and consequent corrections in the experimental work, because 102° F. is a minimum temperature and corrections could be a source of unnecessary error. The temperature can be readily maintained by immersing the lactometer cylinder in a water bath equipped with thermostatic control. However, for workers in the field who might want to use this method where no regulated water bath is at hand, it is suggested that the temperature be allowed to vary not more than two degrees from 102° F. for the reading. For each degree above 102° F., the lactometer reading should be corrected by adding 0.2 lactometer degree, and for each degree below 102° F. it should be corrected by subtracting 0.2 lactometer degree from the reading.

LACTOMETER DESIGN

The preliminary experimental work was carried out with a fragile type of commercial lactometer which was graduated to 0.1 lactometer degree at 60° F., and which had been calibrated by the National Bureau of Standards. When it was used at 102° F., it was necessary to apply a large correction [+ 6.6] for the difference in expansion of glass and milk over the range of 60° to 102° F. Consequently, a new lactometer was designed which is calibrated to read the specific gravity of milk at 102° F., referred to water at 102° F. from 25.6 and 37.4 lactometer degrees.

It was necessary to provide a large bulb in order to obtain an instrument with a sturdy stem and sensitivity of 0.2 degree (two in the fourth decimal place.

expressed as specific gravity). The design was in accordance with the equation for the dimensions of hydrometers derived at the National Bureau of Standards *(15)*. It is possible to read this lactometer to 0.1 degree if desired. A milk sample of about 10 oz. is required. This lactometer is commercially available[2] and the specifications are given in Figure 1.

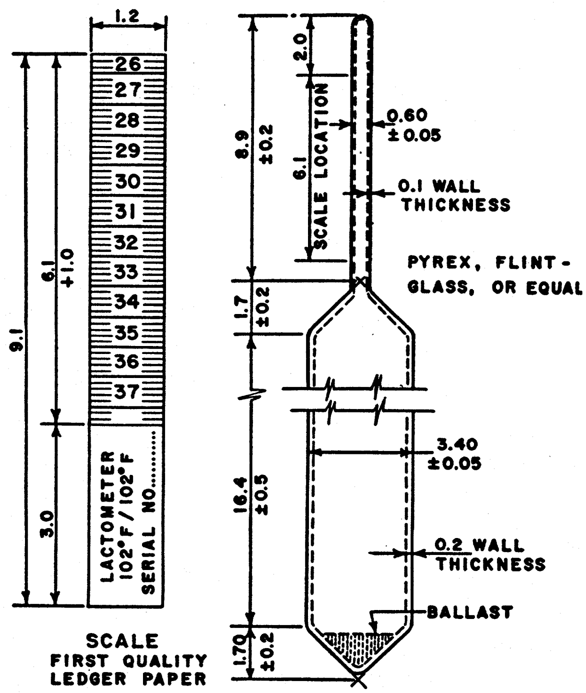


FIG. 1. Dimensions of 102° F. glass lactometer. All dimensions are in centimeters.

It is realized that use of this lactometer for testing the milks of a large number of individual cows in a herd would require much milk. Accordingly, the manufacturer cooperated in designing a smaller experimental model which appears to be suitable (it also has other uses, where the more sensitive instrument is not needed). This small lactometer requires only about 5 oz. of milk and is less susceptible to breakage.

The following dimensions in cm. are approximate, since certain tolerances are allowable for glas hydrometers: total length, 15; stem length, 6.5; stem diameter, 0.5; bulb diameter, 3.5, scale length, 0.23. The bulb displaces about 65 ml. of liquid.

The scale is designed for whole milk over the range of 25.5 to 33.5 degrees, the smallest division being 0.5 degree. Experiments have indicated that readings may be estimated to 0.25 degree.

[2] Catalog No. 22261, Taylor Instrument Companies, Rochester, N. Y. (Mention of this firm does not constitute endorsement of its products by the U. S. Department of Agriculture over similar items or products not mentioned.)

EXPERIMENTAL WORK

The experiments were conducted by rapidly heating the milk samples to about 102° F. in a few minutes in water at about 115° F., with occasional gentle shaking. The samples were then transferred to lactometer cylinders held in a water bath which was thermostatically set at 102° ± 0.3°F. Several readings were made, and in many experiments were checked against other lactometers. Duplicate readings checked to 0.1 lactometer degree. Experiments demonstrated that milk in good condition could be held in cylinders at 102° F. for over one-half hour with a change of only 0.1 degree in the lactometer reading, if gently stirred occasionally.

The Babcock fat tests were made according to the method in general use *(1, 13)*. Accurately calibrated bottles and pipettes were used. The 17.6-ml. pipettes were filled by adjusting the top of the milk surface to the graduation line, then allowed to drain for about 15 seconds before blowing out the last drop. A shaker was used to mix the milk and acid in the test bottles in many of the experiments, and during the summer the acid was cooled prior to use. Readings were made in duplicate and read to hundredths of a per cent fat by means of an illuminated reader with magnifier. Duplicate determinations agreed closely and were often checked by another observer.

Total solids determinations were made in duplicate, using the Mojonnier method and apparatus in which the samples were heated for 10 minutes in a vacuum oven regulated at 100° C. (212° F.) *(14)*. Generally, duplicate results agreed by 0.01 to 0.03% total solids, or closer.

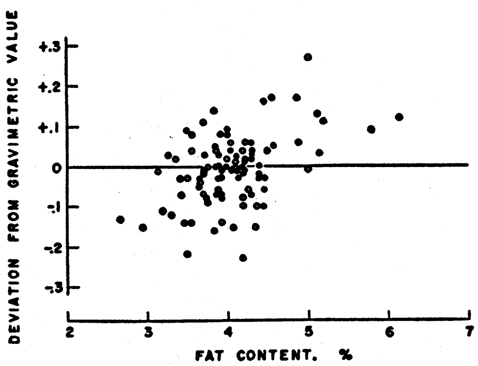
Two hundred samples of milk, selected during all seasons of the year from about 25 herds in Maryland and Virginia, were used in plots of experimental results. The deviations of the percentage of calculated total solids from the percentage of gravimetric total solids were separately plotted for milk samples from two or more cows (see Figure 2) and for milk from individual cows (Figure 3).

The points compromise 101 mixed or herd milk samples and 99 samples from individual cows of six different breeds. The mixed milk experiments included 83 samples from about two to 14 cows and 18 samples from herds and dairy plants which represented from about 15 to 300 cows.

A cooperative research project on this F. lactometer method was conducted by the Dairy Department, College of Agriculture, University of Maryland, and through their courtesy it was possible to include in the calculations the results of 59 tests made on individual cows of the four major breeds *(3)*.[3]

It will be observed that the points indicating the deviation for milk from individual Holstein cows are mostly positive. Not as many cows are represented as would appear, however, since samples from two Holstein cows accounted for 12 of these positive deviations.

[3] This work was sponsored by the American Dairy Association.



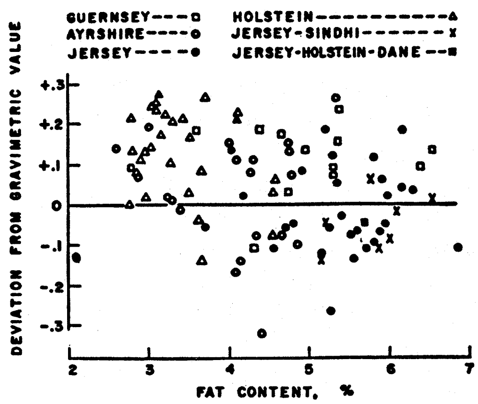


FIG. 2. Deviations of the calculated per cent of total solids from the gravimetric per cent of total solids for milk from two or more cows.

FIG. 3. Deviations of the calculated per cent of total solids from the gravimetric per cent of total solids for milk from individual cows.

Deviations of the calculated values from the gravimetric values for total solids in milk from individual cows were computed and the average deviation was + 0.05%. The standard error of estimate computed from these deviations (about the mean deviation) was $\pm$ 0.13. This measurement can be interpreted as meaning that about two-thirds of the calculated values could be expected to fall within + 0.05 $\pm$ 0.13% of the gravimetric total solids, or within from − 0.08 to + 0.18; about 95% of the calculated values could be expected to fall within + 0.05 $\pm$ 0.26% of the gravimetric values, or within from − 0.21 to + 0.31.

Similarly, for the mixed or herd milk samples, the average deviation was − 0.006% total solids, and the standard error of estimate was $\pm$ 0.091%. Consequently, about 95% of the calculated values on mixed milk could be expected to fall within − 0.006 $\pm$ 0.182% of the gravimetric values, or within from − 0.19 to + 0.18.

A linear least-squares equation was fitted to the gravimetric total solids value for samples from individual cows. This equation for calculating total solids from fat and lactometer values is: $T = 1.364\,F + 0.275\,L - 0.879$. This relationship, calculated from these particular data, agrees well with the theoretically derived equation.

The standard error of estimate of the percentage of total solids computed from this equation was $\pm$ 0.12.

The data for samples of herd milk were represented by the linear least-squares equation: $T = 1.254\,F + 0.271\,L - 0.261$.

This equation had a standard error of estimate of $\pm$ 0.08.

THE METHOD IN PRACTICE

Lactometer Test. Rapidly heat the milk sample (about one-third of a quart) to a temperature of approximately 102° F. by immersing the sample in warm water (about 115° F.). Gently shake the sample at intervals to prevent overheating. The flask should be loosely stoppered, to reduce evaporation.

Transfer the sample with as little agitation as possible to a lactometer cylinder held in a water bath with the temperature thermostatically maintained at 102° F. The lactometer should be preheated for not less than three minutes before using. This can be done by immersing it in a cylinder of water held in in the same constant temperature bath as the milk sample. Remove the lactometer and wipe dry immediately prior to use.

Slowly immerse the lactometer in the milk sample. The reading is taken at the top of the meniscus after the lactometer comes to rest. It is important that the lactometer stem be clean and dry above the milk surface. Repeat readings can be made by withdrawing the lactometer just enough to enable the operator to wipe the stem clean with tissue or a soft cloth before slowly immersing it to the reading point.

Each lactometer degree on the scale is divided into 0.2 degree, and readings may be estimated readily to 0.1 degree. Successive readings should agree by 0.1 degree if the temperature remains constant. The temperature of the milk should be checked with an accurate thermometer at the time of reading.

Fat Test. The fat tests are conducted by the standard Babcock method or its equivalent *(1)*. The surface of the milk in the 17.6-ml. pipette should be adjusted to the graduation line. The amount of fat should be read to the nearest 0.05%. In applying the formula to skimmilk, the fat content was estimated by the American Association test *(5)*, a modification of the Babcock fat test.

General Suggestions. A large number of determinations can be carried out rapidly by using several lactometer cylinders in a constant temperature bath. The cylinders of milk can be held at this temperature for periods of about one hour with only negligible change in the specific gravity, provided that the milk is gently stirred before immersing the lactometer. This long holding method is not suitable for use with old milk, which tends to become rancid or to oil off.

When the milk samples are rather uniform in fat content, it is not necessary, for practical work, to wash and dry the lactometer between each measurement, because the error due to residual milk adhering to the lactometer from one sample to another is negligible *(16)*. However, the instrument should be rapidly transfered and then only the stem need be wiped dry.

Grant *(6)*, in using this lactometer method, found that composite samples could be preserved for eight days with only negligible changes in the specific gravity. The samples were refrigerated at about 40° F. after the addition of 0.5 ml. of formalin (40% solution) per pint of milk.

SUMMARY

A method is presented for the calculation by a formula of the per cent of solids in milk from the percentage of fat, and the lactometer reading made at the the temperature of 102° F.

A lactometer designed for use at 102° F. is described.

The results on 200 samples of milk from about 25 herds show the deviation of the calculated total solids from the gravimetric (Mojonnier) total solids, and are presented graphically. The average deviation was + 0.05% total solids and the standard error of estimate was ± 0.13 for 99 samples of milk from individual cows. The average deviation was − 0.006% total solids and the standard error of estimate was ± 0.09% for 101 samples of mixed or herd milk.

ACKNOWLEDGMENTS

The author wishes to acknowledge the cooperation and assistance of the following individuals: E. O. Whittier, who advised concerning the design of the lactometer and the preparation of the formula; the late G. E. Holm, who made many helpful suggestions; and F. P. Hanrahan, who aided in designing the instrument. L. F. Herrman and Elsie D. Anderson of the Marketing Research Division, Agricultural Marketing Service, cooperated in the statistical analysis of the experimental results. N. E. Yongue of the Chemical Laboratory of the District of Columbia Health Department supplied numerous samples of platform milk from this milk-shed. E. A. Capelle of the Taylor Instrument Companies, Rochester, N. Y., was very helpful in providing experimental models of the new lactometer. C. T. Collett of the Mechanics Division of the National Bureau of Standards gave full cooperation in the calibration of the lactometer.

REFERENCES

(1) Assoc. of Official Agricultural Chemists. *Official Methods of Analysis.* 8th ed., p. 248. Washington, D. C. 1955.

(2) Bakke, A. and Honegger, P. Determination du Poids Specifique du Lait Frais. *Lait,* 3: 3. 1923.

(3) Corbin, E. A. Unpublished data. Eastern Utilization Research Branch, Agricultural Research Service, USDA, Washington, D. C.

(4) Dahlberg, A. C. Variations that May Reasonably Be Expected in Butterfat Tests. *Ann. Rept., N. Y. State Assoc. Dairy and Milk Inspectors,* 6: 191. 1932.

(5) Eckles, C. H., Combs, W. B., and Macy, H. *Milk and Milk Products.* 4th ed., p. 378. McGraw-Hill Book Co., Inc., New York. 1951.

(6) Grant, F. M. Personal communication. Dairy Husbandry Research Branch, Agricultural Research Service, USDA, Beltsville, Md.

(7) Heinemann, B., Cosimini, J., Jack, E. L., Willingham, J. J., and Zakariasen, B. M. Methods of Determining the Per Cent of Total Solids in Milk by Means of the Lactometer. *J. Dairy Sci.,* 37: 869. 1954.

(8) Herrington, B. L. *Milk and Milk Processing.* 1st ed., p. 335. McGraw-Hill Book Co., Inc., New York. 1948.

(9) Herrmann, L. F. Indirect Estimates of the Solids-not-fat Content of Milk. Agricultural Marketing Service, Marketing Research Division, USDA, Washington, D. C. 44 pp. 1954.

(10) Horn, D. W. A Rational Lactometer. *Wagner Free Inst. Sci., Bull.* 10: 1. 1935.

(11) Hoyt, C. F., Smith, N. C., Lampert, L. M., and Saywell, L. G. The Lactometer as Used in the Determination of Solids-not-fat in Milk. *Calif. State Dept. of Agr. Monthly Bull.,* 17: 594. 1928.

(12) Lampert, L. M. Determination of Solids-not-fat by Lactometer Methods. *Proc. 35th Ann. Meeting, Western Div., A.D.S.A.* 1954.

(13) Milk Industry Foundation. *Laboratory Manual. Methods of Analysis of Milk and Its Products.* Washington, D. C. 1949.

(14) Mojonnier, T., and Troy, H. C. *Technical Control of Dairy Products.* p. 122. Mojonnier Bros. Co., Chicago, Ill. 1925.

(15) Peffer, E. L., and Blair, M. G. Testing of Hydrometers. Natl. Bur. Standards *Circ.* 477. 1949.

(16) Roeder, G. Die Fehlerquellen bei der Berechnung des Trockenmassegehaltes der Milch. *Milchwissenschaft,* **8**: 125. 1953.

(17) Sharp, P. S., and Hart, R. G. The Influence of the Physical State of the Fat on the Calculation of Solids from the Specific Gravity of Milk. *J. Dairy Sci.,* **19**: 683. 1936.

(18) Watson, P. D. A Lactometer Method for Determining the Solids in Milk. Eastern Utilization Research Branch, Agricultural Research Service, USDA, Washington, D. C. ARS-73-10. April, 1956.

(19) Whittier, E. O. Determination of Milk Solids. *Proc. 23rd Ann. Inst. Dairying, State College of Washington,* Pullman. 1954.

(20) Ystgaard, O. M., Homeyer, P. G., and Bird, E. W. Determination of Total Solids in Normal and Watered Milks by Lactometric Methods. *J. Dairy Sci.,* **34**: 689. 1951.